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11/18/97 JC549 U.S. PTO

08/972653 11/18/97

PATENT

Attorney's Docket No. 4030C

Box Patent Application
Commissioner of Patents and Trademarks
Washington, D. C. 20231

NEW APPLICATION TRANSMITTAL

Transmitted herewith for filing is the patent application of Inventor(s):

GARY J. PUTERKA, SHEPHERDSTOWN, WV;
DENNIS G. SEKUTOWSKI, STOCKTON, NJ; DAVID MICHAEL GLENN, SHEPHERDSTOWN, WV

For (Title): **METHOD FOR PROTECTING SURFACES FROM ARTHROPOD INFESTATION**

1. Type of Application

This new application is for an

- ☐ Original
- ☐ Design
- ☐ Divisional
- ☐ Continuation
- ■ Continuation-in-part (CIP)

2. Benefit of Prior U.S. Application(s) (35 U.S.C. 120)

■ The new application being transmitted claims the benefit of prior U.S. application(s) and enclosed are ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED.

Certification under 37 CFR 1.10

I hereby certify that this New Application Transmittal request and the documents referred to as attached therein are being deposited with the United States Postal Service on November 18, 1997 in an envelope as "Express Mail Post Office to Addressee" service under 37 CFR 1.10, Mailing Label Number EM361217392 Label Number addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231-0001.

Janet Szot
Janet Szot

3. Papers Enclosed which are required for filing date under 37 CFR 1.53(b) Regular) or 37 CFR 1.153 (Design) Application.

__20__ Pages of specification

__3__ Pages of claims

__1__ Pages of Abstract

_____ Sheets of Drawing

☐ Formal
☐ Informal
☐ The enclosed drawing(s) are photograph(s), and there is also attached a "PETITION TO ACCEPT PHOTOGRAPH(S) AS DRAWING(S)". 37 CFR 1.84(b)

4. Additional papers enclosed:

☐ Preliminary Amendment
☐ Information Disclosure Statement (37 CFR 1.98)
☐ Form PTO-1449
☐ Citations
☐ Other

5. Declaration or oath

☐ Enclosed - signed by inventors

■ Not enclosed.

6. Inventorship Statement

The inventorship for all the claims in this application are:

■ The same
or
☐ Not the Same. An explanation, including the ownership of the various claims at the time the last claimed invention was made,
☐ is submitted
■ will be submitted later.

7. Language -

X English

8. Assignment

☐ an assignment of the invention

☐ is attached. a Separate "cover sheet for assignment document accompanying new patent application, or form PTO 1595 is also attached

■ will follow

9. Certified Copy of Application(s) from which priority is claimed:

_____

_____

☐ **is attached**

☐ **will follow**

10. Fee Calculation (37 CFR 1.16)

A. X Regular application

CLAIMS AS FILED

Number filed	Number Extra		Rate	Basic Fee 37 CFR 1.16(a) **$ 790.00**
Total Claims 9	-20 =	X	22.00	$ -0-
Independent Claims 3	- 3 =	X	82.00	-0-
Multiple dependent claims (if any) 1			270.00	$ 270.00
Total Basic Filing Fee				$1,060.00

11. Fee payment being made at this time

X	Basic Filing Fee	$1,060.00
☐	Recording Assignment	$

12. Charge Account No. <u>05-1070</u> in the amount of $1,060.00

13. Commissioner is hereby authorized to charge any additional fees required by this paper and during the entire pendency of this application to Account No. <u>05-1070.</u>

14. Credit any overpayment to Account No. <u>05-1070.</u>

Reg.No. 28,960

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Signature of attorney
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Iselin, New Jersey 08830

Attorney's Docket No. 4030C **PATENT**

ADDED PAGES FOR APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED

NOTE: "In order for an application to claim the benefit of a prior filed copending national application, the prior application must name as an inventor at least one inventor named in the later filed application and disclose the named inventor's invention claimed in at least one claim of the later filed application in the manner provided by the first paragraph of 35 U.S.C. 112." 37 CFR 1.78(a).

NOTE: "In addition the prior application must be (1) complete as set forth in § 1.51, or (2) entitled to a filing date as set forth in § 1.53(b) and include the basic filing fee set forth in § 1.16; or (3) entitled to a filing date as set forth in § 1.53(b) and have paid therein the processing and retention fee set forth in § 1.21(l) within the time period set forth in § 1.53(d)." 37 CFR 1.78(a).

17. Relate Back

WARNING: *If an application claims the benefit of the filing date of an earlier filed application under 35 U.S.C. 120, 121 or 365(c), the 20-year term of that application will be based upon the filing date of the earliest U.S. application that the application makes reference to under 35 U.S.C. 120, 121 or 365(c). (35 U.S.C. 154(a)(2) does not take into account, for the determination of the patent term, any application on which priority is claimed under 35 U.S.C. 119, 365(a) or 365(b).) For a c-i-p application, applicant should review whether any claim in the patent that will issue is supported by an earlier application and, if not, the applicant should consider canceling the reference to the earlier filed application. The term of a patent is not based on a claim-by-claim approach. See Notice of April 14, 1995, 60 Fed. Reg. 20,195, at 20,205.*

(complete the following, if applicable)

☐ Amend the specification by inserting, before the first line, the following sentence:

A. 35 U.S.C. 119(e)

NOTE: "Any nonprovisional application claiming the benefit of one or more prior filed copending provisional applications must contain or be amended to contain in the first sentence of the specification following the title a reference to each such prior provisional application, identifying it as a provisional application, and including the provisional application number (consisting of series code and serial number)." 37 C.F.R. § 1.78(a)(4).

☐ "This application claims the benefit of U.S. Provisional Application(s) No(s).:

APPLICATION NO(S).:	FILING DATE
____ / ____________	________ "
____ / ____________	________ "
____ / ____________	________ "

B. 35 U.S.C. 120, 121 and 365(c)

NOTE: "Any nonprovisional application claiming the benefit of one or more prior filed copending nonprovisional applications or international applications designating the United States of America must contain or be amended to contain in the first sentence of the specification following the title a reference to each such prior application, identifying it by application number (consisting of the series code and serial number) or international application number and international filing date and indicating the relationship of the applications. Cross-references to other related applications may be made when appropriate. (See § 1.14(b))." 37 C.F.R. § 1.78(2).

☐ "This application is a

- ☐ continuation
- ☒ continuation-in-part
- ☐ divisional

of copending application(s)

☒ application number 08 / 812,301 filed on March 5, 1997 "

☐ International Application _____ filed on

_____ and which designated the U.S."

NOTE: The proper reference to a prior filed PCT application that entered the U.S. national phase is the U.S. serial number and the filing date of the PCT application that designated the U.S.

NOTE: (1) Where the application being transmitted adds subject matter to the International Application, then the filing can be as a continuation-in-part or (2) if it is desired to do so for other reasons then the filing can be as a continuation.

☐ "The nonprovisional application designated above, namely application _____ / _____, filed _____, claims the benefit of U.S. Provisional Application(s) No(s).:

APPLICATION NO(S).:	FILING DATE
_____ /_____	_____ "
_____ /_____	_____ "
_____ /_____	_____ "

NOTE: The deadline for entering the national phase in the U.S. for an international application was clarified in the Notice of April 28, 1987 (1079 O.G. 32 to 46) as follows:

"The Patent and Trademark Office considers the International application to be pending until the 22nd month from the priority date if the United States has been designated and no Demand for International Preliminary Examination has been filed prior to the expiration of the 19th month from the priority date and until the 32nd month from the priority date if a Demand for International Preliminary Examination which elected the United States of America has been filed prior to the expiration of the 19th month from the priority date, provided that a copy of the international application has been communicated to the Patent and Trademark Office within the 20 or 30 month period respectively. If a copy of the international application has not been communicated to the Patent and Trademark Office within the 20 or 30 month period respectively, the international application becomes abandoned as to the United States 20 or 30 months from the priority date respectivley. These periods have been placed in the rules as paragraph (h) of § 1.494 and paragraph (i) of § 1.495. A continuing application under 35 U.S.C. 365(c) and 120 may be filed anytime during the pendency of the international application."

18. Relate Back—35 U.S.C. 119 Priority Claim for Prior Application

The prior U.S. application(s), including any prior International Application designating the U.S., identified above in item 17B, in turn itself claim(s) foreign priority(ies) as follows:

country	appln. no.	filed on

The certified copy(ies) has (have)

☐ been filed on _____, in prior application 0 /_____, which was filed on _____.

☐ is (are) attached.

WARNING: *The certified copy of the priority application that may have been communicated to the PTO by the International Bureau may* ***not*** *be relied on without any need to file a certified copy of the priority application* ***in the continuing application.*** *This is so because the certified copy of the priority application communicated by the International Bureau is placed in a folder and is not assigned a U.S. serial number unless the national stage is entered. Such folders are disposed of if the national stage is not entered. Therefore, such certified copies may not be available if needed later in the prosecution of a continuing application. An alternative would be to physically remove the priority documents from the folders and transfer them to the continuing application. The resources required to request transfer, retrieve the folders, make suitable record notations, transfer the certified copies, enter and make a record of such copies in the Continuing Application are substantial. Accordingly, the priority documents in folders of international applications that have not entered the national stage may not be relied on. Notice of April 28, 1987 (1079 O.G. 32 to 46).*

19. Maintenance of Copendency of Prior Application

NOTE: The PTO finds it useful if a copy of the petition filed in the prior application extending the term for response is filed with the papers constituting the filing of the continuation application. Notice of November 5, 1985 (1060 O.G. 27).

A. ☐ Extension of time in prior application

(This item ***must*** *be completed and the papers filed* ***in the prior application,*** *if the period set in the prior application has run.)*

☐ A petition, fee and response extends the term in the pending **prior** application until _____.

☐ A **copy** of the petition filed in prior application is attached.

B. ☐ Conditional Petition for Extension of Time in Prior Application

(complete this item, if previous item not applicable)

☐ A conditional petition for extension of time is being filed in the pending **prior** application.

☐ A **copy** of the conditional petition filed in the prior application is attached.

20. Further Inventorship Statement Where Benefit of Prior Application(s) Claimed

NOTE: *"If the continuation, continuation-in-part, or divisional application is filed by less than all the inventors named in the prior application a statement must accompany the application when filed requesting deletion of the names of the person or persons who are not inventors of the invention being claimed in the continuation, continuation-in-part, or divisional application." 37 CFR 1.62(a) [emphasis added]. (dealing with the file wrapper continuation situation).*

NOTE: *"In the case of a continuation-in-part application which adds and claims additional disclosure by amendment, an oath or declaration as required by § 1.63 must be filed. In those situations where a new oath or declaration is required due to additional subject matter being claimed, additional inventors may be named in the continuing application. In a continuation or divisional application which discloses and claims only subject matter disclosed in a prior application, no additional oath or declaration is required and the application must name as inventors the same or less than all the inventors in the prior application." 37 CFR 1.60(c) (dealing with the continuation situation).*

(complete applicable item (a), (b) and/or (c) below)

(a) ☐ This application discloses and claims only subject matter disclosed in the prior application whose particulars are set out above and the inventor(s) in this application are

- ☐ the same.
- ☐ less than those named in the prior application. It is requested that the following inventor(s) identified for the prior application be deleted:

_____

(type name(s) of inventor(s) to be deleted)

(b) ☐ This application discloses and claims additional disclosure by amendment and a new declaration or oath is being filed. With respect to the prior application, the inventor(s) in this application are

- ☐ the same.
- ☐ the following additional inventor(s) have been added:

_____

(type name(s) of inventor(s) to be added)

(c) The inventorship for all the claims in this application are

- ☒ the same.
- ☐ not the same. An explanation, including the ownership of the various claims at the time the last claimed invention was made
  - ☐ is submitted.
  - ☐ will be submitted.

21. Abandonment of Prior Application *(if applicable)*

☐ Please abandon the prior application at a time while the prior application is pending, or when the petition for extension of time or to revive in that application is granted, and when this application is granted a filing date, so as to make this application copending with said prior application.

NOTE: According to the Notice of May 13, 1983 (103, TMOG 6-7), the filing of a continuation or continuation-in-part application is a proper response with respect to a petition for extension of time or a petition to revive and should include the express abandonment of the prior application conditioned upon the granting of the petition and the granting of a filing date to the continuing application.

22. Petition for Suspension of Prosecution for the Time Necessary to File an Amendment

WARNING: "The claims of a new application may be finally rejected in the first Office action in those situations where (1) the new application is a continuing application of, or a substitute for, an earlier application, and (2) all the claims of the new application (a) are drawn to the same invention claimed in the earlier application, and (b) would have been properly finally rejected on the grounds of art of record in the next Office action if they had been entered in the earlier application." MPEP, § 706.07(b).

NOTE: Where it is possible that the claims on file will give rise to a first action final for this continuation application and for some reason an amendment cannot be filed promptly (e.g., experimental data is being gathered) it may be desirable to file a petition for suspension of prosecution for the time necessary.

(check the next item, if applicable)

☐ There is provided herewith a Petition To Suspend Prosecution for the Time Necessary to File An Amendment (New Application Filed Concurrently)

23. Small Entity (37 CFR § 1.28(a))

☐ Applicant has established small entity status by the filing of a verified statement in parent application /_____ on _____ .

☐ A copy of the verified statement previously filed is included.

WARNING: "Status as a small entity in one application or patent does not affect any other application or patent, including applications or patents which are directly or indirectly dependent upon the application or patent in which the status has been established. Applications filed as continuations, divisions or continuations-in-part of a parent application must include a reference to a verified statement filed in the parent application if status as a samll entity is still proper and desired." 37 CFR § 1.28(a).

24. NOTIFICATION IN PARENT APPLICATION OF THIS FILING

☐ A notification of the filing of this
(check one of the following)

☐ continuation

☒ continuation-in-part

☐ divisional

is being filed in the parent application, from which this application claims priority under 35 U.S.C. § 120.

METHOD FOR PROTECTING SURFACES FROM ARTHROPOD INFESTATION

Cross-reference to Related Applications

This application is a continuation-in-part of U.S. Patent Application No. 08/812301, filed March 5, 1997, which is incorporated herein by reference for its teachings related to the invention disclosed herein.

Field of the Invention

The present invention is directed to a method for protecting surfaces from arthropod infestation by using certain nontoxic particulate materials.

Background of the Invention

The prior art has discussed the use of certain inert particulate solids as insecticides, see for example; Driggers, B.F., "Experiments with Talc and Other Dusts Used Against Recently Hatched Larvae of the Oriental and Codling Moths," *J. Econ. Ent.*, 22 327-334 (1929); Hunt, C.R., "Toxicity of Insecticide Dust Diluents and Carriers to Larvae of the Mexican Bean Beetle," *J. Econ. Ent.*, 40 215-219 (1947); P. Alexander, J. A. Kitchener and H. V. A. Briscoe, "Inert Dust Insecticides," Parts I, II, and III, *Ann. Appl. Biol.*, 31 143-159, (1944), which concluded that "... the relative killing powers of different dusts run parallel with their capacities for promoting evaporation."; Chiu, S.F., "Toxicity Studies of So-Called 'Inert' Materials with the Rice Weevil and the Granary Weevil," *J. Econ. Entomol.* 32 810-821 (1939); David, W.A.L. and B.O.C. Gardiner "Factors Influencing

the Action of Dust Insecticides," *Bull. Entomol. Res.*, 41 1-61(1950); Ebling, W. and R.E. Wagner, "Rapid Desiccation of Drywood Termites with Inert Sorptive Dusts and Other Substances," *J. Econ. Entomol.* 52 190-207 (1959); Bar-Joseph, M. and H. Frenkel, "Spraying Citrus Plants with Kaolin Suspensions Reduces Colonization by the Spirea Aphid," *Crop Prot.* 2 371-374 (1983); Farmer, A.M., "The Effect of Dust on Vegetation - a Review," *Environ. Pollut.* 79:63-75 (1993); Dahliwal, J.S., "Effect of Rain Fall and Kaolinite Spray on the Corn Leaf Aphid Infesting Barley," *Forage Res.* 5 155 (1979) and US patents 3,159,536 (1964), 3,235,451 (1965), 5,122,518 (1992) and 5,414,954 (1995). In particular '945 relates to "methods and means of selectively controlling the movement of crawling arthropods and more particularly to non-toxic non-debilitating methods and means for causing crawling arthropods to abandon sites they have infested or for discouraging crawling arthropods from infesting a site where they are not wanted." According to '954 this is accomplished by "a method of restricting crawling arthropods from climbing onto a skewed surface comprising forming an adherent, continuous, substantially uniformly thick coating on said surface by applying thereto a dispersion of minus 10 micron titanium dioxide particles in a liquid and '954 specifically refers to a "method wherein said crawling arthropods are cockroaches," each of which is incorporated herein by reference with regard to its teachings relating to particulate materials.

Chemical insecticides have been used extensively in horticultural crop production to control certain arthropod pests such as arthropods and mites. These

chemical insecticides generally belong to the following types of chemical compounds: inorganic (sodium fluoaluminate), organic (dithiocabamates, organophosphates), and antibiotic (agrimectins, spinosins). These chemical insecticides are physiological toxins that kill arthropod pests. Additional insecticidal classes are hormonal (phenoxyphenoxy) that kill arthropods by physiologically disrupting the growth processes, biologicals (entomopathogenic fungi, bacteria, and viruses) that kill by causing fatal diseases, soaps (potassium salts of fatty acids) that kill by suffocation, and diatomaceous earth that kills by desiccation.

The above references refer to particulate solids that are toxic to arthropods and kill these pests.

There is still a need for an effective nontoxic method for protecting surfaces from arthropod infestation based on particulate materials that are not considered harmful to mammals, birds, fish, beneficial arthropods, and the environment.

Summary of the Invention

A method for protecting surfaces from arthropod infestation which comprises applying to said surface an effective amount of one or more particulate materials selected from the group consisting of calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates and mixtures thereof, said particulate materials being finely divided.

Detailed Description of the Invention

This invention relates to a method for protecting surfaces from arthropod infestation. The arthropods controlled by this invention (as well as the damage resulting therefrom) refer to arthropods including insects, mites, spiders and related animals. This invention is particularly effective in controlling damage caused by crawling, hopping or flying arthropod pests without using insecticides or physiological toxins that are harmful to human health and the environment. In addition, the method of this invention is non-toxic to many beneficial arthropods such as lady beetles and honey bees. Inclusive are other types of damage to crops commonly caused by arthropod transmission of disease such as the fungus disease, Dutch Elm disease, of American Elm by the European elm beetle; the bacterial disease, Fire blight, of apples and pears by flies, beetles and other insects; the virus disease, Curly Top, of sugar beats by the beet leaf hopper. Damage control also applies to those secondary infections of wound sites on a plant that result from arthropod feeding such as brown rot infection of stone fruits that results when the disease organism enters the plant through plum curculio feeding sites.

The instant invention provides a nontoxic environment that is effective at protecting surfaces against a broad-spectrum of arthropod pest activity by being effective against arthropods that crawl, hop and fly. Although some arthropods may die as a result of contacting the particles of this invention, the primary function of the treatment of this invention is to affect arthropod behavior instead of killing the arthropod and,

therefore is not considered harmful to many beneficial insects such as ladybugs and honeybees. While not being bound by theory, the effects of the particle treatment of this invention protect the surface by creating a hostile environment on the surface that repels arthropod pests such that they will not feed, lay eggs, or colonize and, therefore, will not infest or will abandon the treated site by making the surface become unrecognizable by feel, sight or otherwise and/or unpalatable or otherwise unsuitable as a food source or for colonization by arthropod pests failure to recognize the particle-altered surface or otherwise. These effects will vary by arthropod species and size. The particulate treatment does not need to have a smooth surface or one that is set at least a 20 degree angle to the horizon or a continuous coating free of bubbles and voids. A complete coating of the surface is desirable although certain gaps and voids are expected but will not influence the overall arthropod controlling feature of the treatment. The particles useful for this invention can be applied to surfaces that are horizontal or inclined, smooth or rough, or complex or simple in structure and a continuous bubble and void free film is not required for the particle treatment to be effective against most arthropods.

The surfaces to which this invention relate include surfaces that are subject to arthropod infestation and include, for example, man-made structures made of wood, concrete, plastic pipe, electrical cable etc. and include household applications such as protecting plumbing, clothes closets, food cabinets, electrical wiring, foundation, framing, basements, etc.; livestock; soils

including rangeland; stored agricultural products such as grains, seeds etc.; and agricultural and ornamental crops and the products thereof, including those selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants.

The particulate materials useful for the purposes of this invention are selected from the group consisting of calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates and mixtures thereof.

Calcined kaolin is will known to those of ordinary skill in the art and can be prepared by calcining hydrous kaolin which is generally represented by the formula $Al_4Si_4O_{10}(OH)_8$. The calcined kaolin of this invention will usually have been subject to calcination temperature conditions in excess of about 350°C, more typically in excess of about 500°C and preferably between about 500°C and about 1100°C.

Calcium carbonate is a commonly available material. It occurs in nature as, for example, aragonite, calcite, chalk, dolomite, limestone, etc. or may be prepared synthetically by precipitation from the reaction of calcium chloride and sodium carbonate in water or by passing carbon dioxide through a suspension of hydrated lime in water.

Calcined kaolins, hydrous kaolins, and calcium carbonates are normally hydrophillic but their surfaces can be made hydrophobic by addition of hydrophobic wetting agents. Many industrial mineral applications, especially in organic systems such as plastic composites,

films, organic coatings or rubbers, are dependent upon just such surface treatments to render the mineral surface hydrophobic; see, for example, Jesse Edenbaum, Plastics Additives and Modifiers Handbook, Van Nostrand Reinhold, New York, 1992, pages 497-500 which is incorporated herein by reference for teachings of such surface treatment materials and their application. So-called coupling agents such as fatty acids and silanes are commonly used to surface treat solid particles as fillers or additives targeted to these industries. Such hydrophobic agents are well known in the art and common examples include: organic titanates such as Tilcom® obtained from Tioxide Chemicals; organic zirconate or aluminate coupling agents obtained from Kenrich Petrochemical, Inc.; organofunctional silanes such as Silquest® products obtained from Witco or Prosil® products obtained from PCR; modified silicone fluids such as the DM-Fluids obtained from Shin Etsu; and fatty acids such as Hystrene® or Industrene® products obtained from Witco Corporation or Emersol® products obtained from Henkel Corporation (stearic acid and stearate salts are particularly effective fatty acids and salts thereof for rendering a particle surface hydrophobic).

The term "hydrophobic" as used herein with respect to particulate materials of calcined kaolins, hydrous kaolins, and calcium carbonates shall mean that the surface of such particles are made hydrophobic by addition of hydrophobic wetting agents as described hereinabove.

Examples of preferred particulate materials suitable for the purposes of this invention that are commercially

available from Engelhard Corporation, Iselin, NJ are the hydrous kaolins sold under the trademark ASP®, calcined kaolins sold under the trademark Satintone® and the siloxane treated calcined kaolins sold under the trademark Translink®; and calcium carbonate commercially available from English China Clay under the trademarks Atomite® and Supermite® and stearic acid treated ground calcium carbonates commercially available from English China Clay under the trademarks Supercoat® and Kotamite®.

The term "finely divided" when utilized herein means that the particulate materials have a median individual particle size below about 10 microns and preferably below about 3 microns and more preferably the median particle size is about one micron or less. Particle size and particle size distribution as used herein are measured with a Micromeritics Sedigraph 5100 Particle Size Analyzer. Measurements were recorded in deionized water for hydrophilic particles. Dispersions were prepared by weighing 4 grams of dry sample into a plastic beaker adding dispersant and diluting to the 80 ml mark with deionized water. The slurries were then stirred and set in an ultrasonic bath for 290 seconds. Typically, for kaolin 0.5% tetrasodium pyrophosphate is used as a dispersant; with calcium carbonate 1.0% Calgon T is used. Typical densities for the various powders are programmed into the sedigraph , e.g., 2.58 g/ml for kaolin. The sample cells are filled with the sample slurries and the X-rays are recorded and converted to particle size distribution curves by the Stokes equation. The median particle size is determined at the 50% level.

Preferably, the particulate material has a particle size distribution wherein up to 90% by weight of the particles have a particle size of under about 10 microns, preferably below about 5 microns and more preferably about one micron or less.

The particulate materials particularly suitable for use in this invention are nontoxic.

The particulate materials are preferably nontoxic meaning that they are not physiological toxins and, in the limited quantities needed affect arthropod behavior to reduce arthropod infestation, such materials are not considered harmful to mammals, birds, and fish as well as well as arthropods, the environment, the applicator and the ultimate consumer.

This treatment when applied to horticultural crops should not materially affect the exchange of gases on the surface of said crop. The gases which pass through the particle treatment are those which are typically exchanged through the surface skin of living plants. Such gases typically include water vapor, carbon dioxide, oxygen, nitrogen and volatile organics.

The surface to be protected is treated with an amount of one or more particulate materials selected from the group consisting of calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates and mixtures thereof, that is effective in protecting the surface from arthropod infestation. The treatment coverage of said surface is within the skill of the ordinary artesian. Less than full surface coverage is within the scope of this invention and can be highly

effective, for example, with respect horticultural crops neither the under surface of the crop (that which is not exposed directly to the source of light) need be treated by the method of this invention nor must the upper surface of the crop be completely covered; although full crop coverage can provide additional benefits such as effective disease control, smoother fruit surface, reduced bark and fruit cracking, and reduced russetting. Reference is made to U.S. Serial No. ____________, filed concurrently herewith on _________, entitled "Treated Horticultural Substrates" which is incorporated herein by reference for its teachings regarding methods for achieving these additional benefits. The method of this invention may result in the residue of the treatment forming a membrane of one or more layers of said particulate materials on the surface to be treated.

The particulate materials useful for the purposes of this invention may be applied as a dust or as a slurry of finely divided particles in a volatile liquid such as water, a low boiling organic solvent or low boiling organic solvent/water mixture. Adjuvants such as surfactants, dispersants or spreaders/stickers (adhesives) may be incorporated in preparing an aqueous slurry of the particulate materials of this invention. One or more layers of this slurry can be sprayed or otherwise applied to the surface. The volatile liquid is preferably allowed to evaporate between coatings. The residue of this treatment may be hydrophilic or hydrophobic. Applying particles as a dust may be achieved by sprinkling, pouring, or dusting said particles directly on the surface to be protected as an

alternative method for carrying out the method of this invention.

Surfactants that are anionic, cationic or nonionic materials; and/or spreader/stickers that can be mixed with the particles useful for this invention (3% or more solids in water) to aid in spraying uniform treatments on the surfaces to be treated are: modified phthalic glycerol alkyd resins such as Latron B-1956 from Rohm & Haas Co.; Plant oils such as cotton seed oil, or plant oil based materials (cocodithalymide) with emulsifiers such as Sea-wet from Salsbury lab, Inc. or ; Polymeric terpenes such as Pinene II from Drexel Chem. Co.; nonionic detergents (ethoxylated tall oil fatty acids) such as Toximul 859 and Ninex MT-600 series from Stephan.

The particle treatment may be applied as one or more layers of finely divided particulate material. The amount of material applied is within the skill of one of ordinary skill in the art. The amount will be sufficient to repel or otherwise affect arthropod behavior and/or colonization on the surface to which these particles are applied. For example, this can typically be accomplished by applying from about 25 up to about 5000 micrograms of particulate material/cm^2 of surface for particles having specific density of around 2-3 g/cm^3, more typically from about 100 up to about 3000 and preferably from about 100 up to about 500. In addition, environmental conditions such as wind and rain may reduce coverage of the particulate materials on the protected surface and, therefore, it is within the scope of this invention to apply the said particles to the surface being protected

one or more times so as to maintain the desired effect of invention.

The low boiling organic liquids useful in the present invention are preferably water-miscible and contain from 1 to 6 carbon atoms. The term "low boiling" as used herein shall mean organic liquids which have a boiling point generally no more than 100°C. These liquids enable the particulate solids to remain in finely divided form without significant agglomeration. Such low boiling organic liquids are exemplified by: alcohols such as methanol, ethanol, propanol, i-propanol, i-butanol, and the like, ketones such as acetone, methyl ethyl ketone and the like, and cyclic ethers such as ethylene oxide, propylene oxide and tetrahydrofuran. Combinations of the above-mentioned liquids can also be employed. Methanol is the preferred low boiling organic liquid.

Low boiling organic liquids may be employed in applying the particles to surfaces for the purposes of this invention. Typically, the liquids are used in an amount sufficient to form a dispersion of the particulate material. The amount of liquid is typically up to about 30 volume percent of the dispersion, preferably from about 3 up to about 5 volume percent, and most preferably from about 3.5 to about 4.5 volume percent. The particulate material is preferably added to a low boiling organic liquid to form a slurry and then this slurry is diluted with water to form an aqueous dispersion. The resulting slurry retains the particles in finely divided form wherein most of the particles are dispersed to a particle size of less than about 10 microns.

The following examples are illustrative of embodiments of the invention and are not intended to limit the invention as encompassed by the claims forming part of the application.

Example I

Acute toxicity of a hydrophillic kaolin made hydrophobic by treatment with siloxane, Translink® 77, on adult honey bees. Percent mortality was determined 48 hours after exposure to different concentrations of kaolin solubilized in 2 μl of methanol. Mortalities were compared to an untreated control and solvent control. Applications were made topically to 20 adult bees per treatment with 3 replications per treatment. Data is a summary of an acute honey bee toxicity test conducted by Wildlife International, LTD. (Proj. No. 469-101) for Engelhard Corporation.

Table I

Honey bee mortality 48 hours after Translink® 77

Treatment	Dose (μg a.i./bee)	% Mortality
Untreated	none	0.3
Solvent control	2 μl methanol	0.0
Translink 77	6.25.	0.0
	12.5	0.0
	25.0	0.3
	100.0	0.0

Translink® 77 siloxane treated kaolin(Engelhard Corporation).

This study shows that Translink® 77 is nontoxic to honey bees at a broad range of concentrations.

Example II

Acute toxicity of a hydrophillic kaolin made hydrophobic by siloxane treatment, Translink® 77, on lady beetle adults as compared to untreated control and toxic conventional insecticide.

Applications were applied 25 pounds material suspended in 4 gal methanol and added to 100 gal water. These treatments were applied at the total output of 125 gal/acre using an orchard handgun sprayer. There were 5 replications per treatment with single tree replicates. Lady beetle mortality was determined by counting the number of dead lady beetles on the ground in a 3 foot diameter around the base of each treated tree. Detrimental effect of applications on live lady beetle numbers within treated trees was determined by counting total number within each treated tree. Data was analyzed using ANOVA and means were compared using the least significant differences method, LSD, at $P = 0.05$.

Table II

Mean (±SE) number of live lady beetle adults within the tree and dead lady beetles on the ground around each treated tree in a pear orchard 2 days after treatment, August 8, 1997, Kearneysville, WV.

Treatment	Concentration	No. Lady beetles/tree	Dead lady beetles on ground/tree
Translink® 77 Kaolin	0.3% in H_2O	3.8 ± 1.1a	0.0 ± 0.0b
Agrimek (avermectin)	5.0 oz. a.i./acre	1.4 ± 0.7b	3.6 ± 0.7a
Untreated control	-	5.4 ± 0.9a	0.0 ± 0.0b

Means within a column followed by the same letter are not significantly different (P > 0.05, LSD).

Data indicates that siloxane treated hydrophobic kaolin particle, Translink® 77, was not harmful to lady beetle populations within trees treated with this compound in comparison to the untreated control. Furthermore, lady beetles were not killed by the Translink® 77 treatment although the toxic chemical, Agrimek®, did. This study shows that Translink® 77 is nontoxic to beneficial lady beetles.

Example III

This example demonstrates how kaolin and calcium carbonate particle barriers are repellent and/or deterrent to egg laying by pear psylla. Ten adults were given a free choice between pear leaves treated with various types of calcium carbonate and kaolin particles that are hydrous, calcined or made hydrophobic by treatment with siloxane or stearate. Leaves were sprayed with a solution comprised of 5% particles and 10%

methanol in water using a hand held sprayer. Treatments included untreated and a 10% MEOH controls. Five mating pairs of adult pear psylla (n = 10) were released within a caged arena containing all eleven particle and untreated control treatments. The experiment was a randomized block design with 5 replications. Adult and egg numbers were recorded 24 hours after being released within the arena. Data was subjected to ANOVA and means were separated using LSD, $P = 0.05$.

Table III

Repellant and oviposition deterrent effects of pear leaves treated with kaolin and calcium carbonate particle treatments on pear psylla adults.

Particle type	Treatment	Number present 24 hours after exposure	
		Adults	Eggs
--	Control	3.60 ± 1.47 AB	15.8 ± 7.69 A
--	Methanol Control	4.40 ± 0.51 A	9.40 ± 4.24 AB
Kaolin	ASP 900- hydrous[1]	5.20 ± 1.24 A	7.20 ± 5.50 BC
	ASP 900- hydrophobic[2]	1.00 ± 0.77 CDE	0.00 ± 0.00 C
	Satintone-W - calcined[3]	2.40 ± 0.60 BC	0.00 ± 0.00 C
	Satintone-W - hydrophobic[4]	1.00 ± 0.45 CDE	0.00 ± 0.00 C
	Translink 37- hydrophobic[5]	0.00 ± 0.00 E	0.00 ± 0.00 C
	Translink 77- hydrophobic[5]	0.40 ± 0.40 DE	0.00 ± 0.00 C
Calcium carbonate	Kotomite - hydrophobic[6]	0.00 ± 0.00 E	0.00 ± 0.00 C
	Atomite - hydrophyllic[7]	1.80 ± 0.58 BCDE	0.00 ± 0.00 C

1. ASP® 900 (Engelhard Corporation) 2. ASP® 900 (Engelhard Corporation) treated with stearate. 3. Satintone® W (Engelhard Corporation) 4. Satintone® W (Engelhard Corporation) treated with stearate 5. Translink® 37 and 77 (Engelhard Corporation) 6. Kotamite® (ECC Int.) 7. Atomite® (ECC Int.)

Means within a column followed by the same letter are not significantly different, LSD, $P = 0.05$; mean of 5 replications.

Results demonstrate that these particles are repellent to adults in that they will not settle upon hydrophobic or calcined kaolin or hydrophobic calcium carbonate particle treated pear leaves. Data indicate that the repellent nature of these particles increases when hydrophillic materials are calcined or when hydrophillic materials are made hydrophobic.

Example IV

"Red Delicious" apple trees received the following treatments: 1) no treatment, 2) weekly application of siloxane treated hydrophobic kaolin particle, Translink® 77, beginning in March 11, 1997, 3) weekly applications of calcined hydrophillic kaolin particle, Satintone® 5HB, beginning in April 29,1997, and 4) weekly application of stearate treated hydrophobic calcium carbonate, SuperCoat®,(commercially available from English China Clay) beginning in April 29, 1997. Kaolin and calcium carbonate treatments were applied 25 pounds material suspended in 4 gal methanol and added to 100 gal water. Satintone® 5HB applied 25 pounds material suspended in 100 gal water with the addition of 27oz Ninex® MT-603 and 2 pints Toximul®. These treatments were applied at the total output of 125 gal/acre using an orchard sprayer. The treatments were arranged in a randomized complete block design with 4 replications and 3 trees/plot. Treatments were not irrigated and received 21.58 cm of precipitation from 1 May to 30 August 1997. Fruit were harvested at

maturity; fruit number were measured at harvest. Data were analyzed using Analysis of Variance (ANOVA) and treatment means were separated using least significant difference method (LSD) at $P = 0.05$.

Table IV

Mean (±) arthropod numbers per terminal in various treatments on 'Red Delicious' apples on 7/1/97. There were 4 replications per treatment and 25 terminals per replication, Kearneysville, WV.

Arthropod	Untreated	Translink 77 Kaolin	Satintone 5HB Kaolin	**SuperCoat Calcium Carb.**
Pests				
Mites	67.5±27.2 a	1.5±0.9 b	3.8±1.4 b	45.8±18.2 a
Leafhoppers	61.0±16.0 a	8.0±5.2 b	16.5±2.3 b	44.8±7.7 a
Thrips	4.5±1.9 a	0.5±0.5 b	1.0±0.6 b	3.0±0.7 ab
Leaf Miners	0.0±0.0 a	1.0±0.6 a	0.5±0.3 a	0.8±0.5 a
Beneficials				
Lacewing Eggs	1.8±0.6 ab	2.3±0.5 ab	0.8±0.8 b	0.3±0.3 b
Lacewing Larvae	0.0±0.0 a	0.3±0.3 a	0.5±0.3 a	0.0±0.0 a
Lady Beetle Larvae	0.0 ± 0.0 b	1.3±1.0 b	4.8±2.2 a	0.3±0.3 b
Lady Beetle Adult	0.0±0.0 a	0.5±0.5 a	0.8±0.5 a	0.3±0.3 a

Means within a column followed by the same letter are not significantly different, LSD, $P = 0.05$.

After 10 applications of the kaolin treatments gave the same or better levels of control of mites, leafhoppers, and thrips. The calcium carbonate treatment gave the same levels of control as kaolin treatments except for mites and leafhoppers. The kaolin and/or calcium carbonate treatments either did not significantly lower or increased beneficial arthropod numbers as compared to the untreated control block. No plant toxicity was noted in any treatment.

Conclusion from this data is that the kaolin and/or calcium carbonate treatments are effective against a broad range of arthropod pests without being harmful to beneficial arthropods.

Example V

Arthropod pest control in blackberries after 6 weekly applications of siloxane treated hydrophobic kaolin particle, Translink® 77, as compared to an untreated control. Applications were treatments were applied at 25 pounds material suspended in 4 gal methanol and added to 100 gal water. These treatments were applied at the total output of 125 gal/acre using an orchard sprayer. There were 3 replications per treatment. Arthropod counts were determined as average numbers per terminal from 10 terminals per treatment. Counts were made on June 24, 1997. Data were analyzed using Analysis of variance and treatment means were separated using least significant difference method (LSD) at $P = 0.05$.

Table V

Mean(±SE) numbers of arthropods per terminal after 6 weekly applications of Translink® 77 kaolin on blackberry, June 24, 1997, Kearneysville, WV

	Untreated	Translink® 77 Kaolin
Leafhoppers	57.8 ± 9.2 a	0.7 ± 0.3 b
Thrips	3.5 ± 0.9 a	0.7 ± 0.3 b
Aphids	7.2 ± 2.4 a	0.7 ± 0.5 b
Psylla Damage[1]	20.7 ± 4.6 a	0.8 ± 0.5 b

Means within a column followed by the same letter are not significantly different, LSD, $P = 0.05$.

[1]Numbers represent whole block counts.

Data demonstrates that siloxane treated hydrophobic kaolin particles, Translink® 77, are very effective control agents against a broad range of arthropod pests of blackberry.

WHAT IS CLAIMED IS:

1. A method for protecting surfaces from arthropod infestation which comprises applying to said surface an effective amount of one or more particulate materials selected from the group consisting of calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates and mixtures thereof, said particulate materials being finely divided.

2. The method of claim 1 wherein the particulate material has a particle size distribution wherein up to 90% of the particles have a particle size of under about 10 microns.

3. The method of claim 1 wherein said hydrophobic calcined kaolins, hydrophobic hydrous kaolins, and hydrophobic calcium carbonates have a hydrophobic outer surface prepared from materials selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

4. The method of claim 1 wherein the surface is a horticultural crop selected from agricultural and ornamental crops.

5. The method of claim 4 wherein the horticultural crop is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

6. The method of claim 1 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

7. A method for protecting horticultural crops from arthropod infestation which comprises applying to the surface of a horticultural crop selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants which comprises applying to the surface of said horticultural crop an effective amount of a slurry of one or more particulate materials selected from the group consisting of calcium carbonate, hydrophobic hydrous kaolin, calcined kaolin, and mixtures thereof, said particulate materials have a median individual particle size of about one micron or less, and wherein said particles as applied allow for the exchange of gases on the surface of said crop.

8. The method of claim 1 or 7 wherein the finely divided particulate materials are applied one or more times during the growing season of said horticultural crop.

9. A method for protecting surfaces from arthropod infestation which comprises applying to the surfaces of agricultural products, man-made structures, and soils, an effective amount of one or more particulate materials selected from the group consisting of calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates and mixtures thereof, said particulate materials being finely divided.

ABSTRACT OF THE DISCLOSURE

Disclosed is a method for protecting surfaces from arthropod infestation which involves treating the surface with an effective amount of finely divided calcined kaolins, hydrophobic calcined kaolins, hydrous kaolins, hydrophobic hydrous kaolins, hydrophobic calcium carbonates, calcium carbonates or mixtures thereof.